



DECLARATION UNDER 37 C.F.R. §1.131



I, Richard O. Ruhr, attest and say as follows:

1. I have been employed by Ecolab, Inc. in Research & Development since September, 1987. My current job title is Principal Chemist. I am a Project Leader in the formulation of cleaning products for industrial applications in the food and beverage markets, experimental design and laboratory performance evaluation of products, field test evaluation of product performance, research in development of cleaning product innovation, product specification creation, preparation of marketing launch information, product label creation, dispensing system design and evaluation, formulation of hand care products, am involved in studies for FDA regulated hand care products.
2. Prior to February 13, 2001, I was involved with the development of new and improved cleaning formulations and methods, and was specifically involved with the experimentation leading to the above-referenced patent application.
3. I am a co-inventor of the subject matter of the claims of the above-referenced U.S. Patent Application Serial No. 09/839, 342.
4. Exhibits A, B and C which are enclosed herewith are true and accurate copies of portions of a Record of Invention marked as Exhibit A and laboratory notebook pages including descriptions of pre-rinse compositions prepared in accordance with the invention disclosed and claimed in the patent application identified above. This portion of the Record of Invention and laboratory notebook pages are provided in support of conception and reduction to practice of the inventive method compositions employed therein described and claimed in the above-referenced patent application prior to February 13, 2001.
5. The laboratory notebook pages 10465-1, 10465-3 and 10465-7, marked as Exhibit B, illustrate testing conducted with a base pre-rinse composition noted as KX-2100 which

is shown on page 3 of the Record of Invention marked as Exhibit A. Variations of the composition were tested by removing one raw material at a time to determine what effect, if any, each raw material had on the cleaning ability of the composition. As shown from these pages, the composition without any partially neutralized polyacrylate, exhibited 80% of the gross soil remaining after 15 minutes. The partially neutralized polyacrylate was maintained in all of the other compositions which exhibited much improved soil removal compared to the formula without any polyacrylate. *- not shown*

Laboratory notebook page 10465-7 illustrates testing with KX-2100 which exhibited essentially total soil removal.

6. Exhibit C includes laboratory notebook pages 10405-93, 10405-105, 10405-107, 10405-129 and 10405-131. Soil panels were prepared and then different rinse compositions tested. Soil panel preparation is described on pages 93, 105 and 107. Testing of a partially neutralized polyacrylate, Paradigm 2030, as compared to alkaline composition which is a standard in the industry, and soft water only, is shown on pages 129 and 131. While the Paradigm 2030 exhibited almost complete soil removal, the alkaline pre-rinse industry standard, shown at 12, exhibited about 5% soil remaining on the panel (page 131).

All statements made herein of my knowledge are true and all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code and that such willful false statements may jeopardize the validity of the application or any patent issued thereon.

Respectfully submitted,

Richard O. Rubin

Address
City

Richard O. Ruhr

Richard O. Ruhr

2864 Colbert Avenue NW
Buffalo, Minnesota 55313
Citizen of the United States of America

Date: 5-22-03

CONFIDENTIAL/ATTORNEY-CLIENT PRIVILEGED

Please route this form to the following individuals:

1. Technical Director

Shaun Kennedy *SK*

2. ATTENTION: A. D. Sorensen
Research Center

FOR OFFICE USE ONLY

Date: _____

Ecolab Code: 1394.00

RECORD OF INVENTION (ROI)

ECOLAB INC.
Research Center
St. Paul, Minnesota 55118

DO NOT LEAVE BLANKS. ENTER NONE IF THE STATEMENT DOESN'T APPLY. PLEASE TYPE OR PRINT. MARK ANY ATTACHMENTS TO INDICATE THE QUESTION TO WHICH THEY RELATE.

TITLE: Process and Composition for Removing Mineral and Protein Soils

INVENTOR(S): Rick O. Ruhr, Joseph I. Kravitz and Andrew T. Gioino

KEYWORDS (for indexing computer files):

Cleaning, Protein, Mineral, Polyanionic Polymer

The purpose of this document to convey to Ecolab's Patent Counsel an inventive idea. It has been prepared pursuant to the request of Ecolab's Patent Counsel.

WHAT DID YOU DO?

- a. What is the name of the research and development project that led to the invention?

Single-Phase Acid Cleaning

- b. What are the overall goal(s) of the project?

Clean protein soiled heat transfer surface with no or reduced amounts of caustic.

- c. Which goal of the research and development project led to the work underlying the technology development disclosed in this ROI?

Cleaning with no caustic

- d. What development in this research and development project is the focus of this ROI?

Use of a polyanionic polymer as a pre-rinse aid in the first step of a multi-step cleaning program

- e. Each technology development can be embodied in a chemical composition, a process for making or using such a chemical composition, or in a device for making or using such a composition. Please list one or more *representative chemical composition(s), method(s) of making or using the chemical composition or device(s) for making or using the composition*. Make sure to include all uses for all divisions of Ecolab.

Cleaning a complex soil comprising protein and/or mineral, such as those found on heat transfer surfaces that process dairy and food products, typically involves a multi-step cleaning program that begins with a water pre-rinse to remove the gross or loosely adherent soil. The use of a water soluble, polyanionic polymer in the water pre-rinse greatly enhances the removal of these gross soils. Adding a polymer containing partially neutralized acid groups to the initial water pre-rinse can be embodied in the following composition:

Partially neutralized sodium polyacrylate	0.0005 – 5.00%
Water	99.9995 – 95.00%

This process is not meant to be limited to food soils found on heat transfer surfaces, but is useful for cleaning any complex soil containing mineral and protein where the initial step is a water pre-rinse. Examples of such soils would be in the laundry area for the removal of food, blood, urine, fecal and perspiration stains. This process could also be useful in warewashing where the soil is not necessarily heat set, but the first step in an automatic dishwasher is a water pre-rinse. One can also see the utility in cleaning hard surfaces such as counters, tiles and bathroom fixtures as well as cleaning surgical instruments where a water pre-rinse or soak may be applied.

The composition is readily prepared by adding the polymer in a concentrated form (liquid or solid), which can be in either an unneutralized or partially neutralized form, to a sufficient amount of water to supply the desired concentration at use. If the concentrated form of the polymer is unneutralized or not neutralized to the desired extent, then the water must contain an amount of neutralizing agent, such as sodium hydroxide, to achieve the desired level of neutralization at use.

HOW DID YOU DO IT?

- a. Describe the circumstances surrounding the creation of the first inventive concept.

Looked at enzymes in a base composition that contained potassium hydroxide, EDTA, potassium carbonate and partially neutralized polyacrylic acid for the removal dairy soils from heat transfer surfaces. Turned out that the desired effect was achieved by the polyacrylic acid within an optimum pH range. Presumably, the pH dictates the degree of neutralization of the polymer.

- b. Please attach to this ROI or insert here *graphs, tables of data, examples* or other research and development information that provides a basis for demonstrating that each goal set for this research and development has been achieved and any surprising results are demonstrated.

See attached lab notebook pages. This ROI is based on subjective observations that show conspicuous differences when compared to appropriate controls.

- c. What is the *best* formula, sequence of method steps or equipment embodiment known to you *at this time*?

The following composition is diluted with water so that its use dilution is 600 ppm by weight and the pH of the use solution is 9.5-10.0.

Item	Percent	RM Code	Raw Material
10	50.60000	100032	WATER DEIONIZED
20	0.20000	250126	TETRASODIUM EDTA LIQ. 40%,
30	13.20000	114009	POTASSIUM HYDROXIDE, 45% LIQUID
40	19.50000	250324	POLYACRYLIC ACID (50%), PARTIALLY NEUTRALIZED (5-10% neutralized)
50	16.50000	113001	POTASSIUM CARBONATE, DENSE GRANULAR

Total	100.00000		

Add items in order listed stirring in between until homogeneous.

HOW DID YOU DO IT?

- d. For every composition, component or step, please provide an *exact chemical name*, as well as a trade name or other code; product identification; or product information data sheet to explain in understandable terms the nature of the chemical or part.

CAS No.	RM Code	Raw Material
64-02-8	250126	Ethylenediamine tetraacetic acid, tetrasodium, salt (40% solution)
1310-58-3	114009	POTASSIUM HYDROXIDE (45% solution)
9003-04-7	250324	POLYACRYLIC ACID (50% solution), PARTIALLY NEUTRALIZED (5-10% neutralized)
584-08-7	113001	POTASSIUM CARBONATE, DENSE GRANULAR

- e. For every test performed in evaluating the inventive concept, please provide an ASTM, AOAC or other *test procedure* reference number. If no reference number is available, please attach the relevant Ecolab test procedure.

See lab notebooks- pages attached

A weighed amount of condensed whole whey (2 – 4 gm) was brushed on to pre-cleaned 2 x 4 inch SS steel (316) polished panel. The panels were steamed to heat set the soil by placing them in a pressure cooker (max 5 lbs pressure) over 200 g water and held at about 138 F for 4.25 hr by heating the pressure cooker on a hotplate set at 215 F plate temperature.

Cleaning observations were made by soaking panels (no agitation) at 140 F in 300 gm of appropriate test solutions and making observations over time.